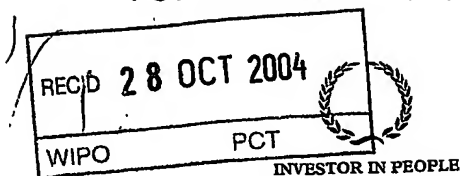




GB04104373



The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

PRIORITY DOCUMENT

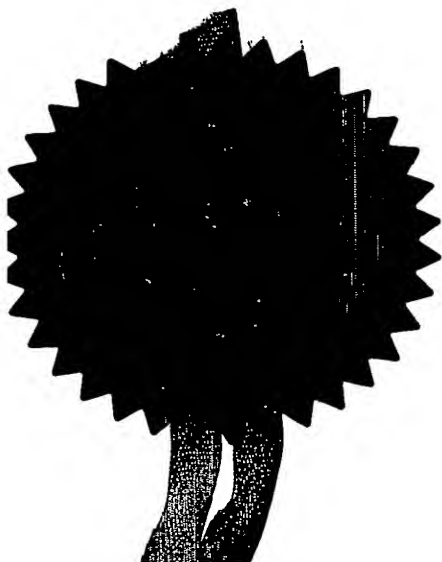
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



Signed *Andrew Gersey*
Dated 6 October 2004

BEST AVAILABLE COPY

The
Patent
Office

1/77

Request for grant of a patent

See the notes on the back of this form. You can also get
an explanatory leaflet, from the Patent Office to help
you fill in this form)

THE PATENT OFFICE
C

16 OCT 2003

NEWPORT

CDK2133

The Patent Office

Cardiff Road
Newport

South Wales NP10 8QQ

16 OCT 03 ED45017 11 002000
P01/7700 0.00-0324238.5

Your reference

Patent application number
(The Patent Office will fill in this part)

0324238.5

16 OCT 2003

Full name, address and postcode of the or of
each applicant (underline all surnames)

RHODIA CONSUMER SPECIALTIES LIMITED
OAK HOUSE
REEDS CRESCENT
WATFORD
HERTS WD24 4QP

Patents ADP number (if you know it)

8486797001

If the applicant is a corporate body, give the
country/state of its incorporation

GB AL 17/10/03

1. Title of the invention

FORMULATION FOR CORROSION AND SCALE
INHIBITION

5. Name of your agent (if you have one)

Barker Brettell

"Address for service" in the United Kingdom
to which all correspondence should be sent
(including the postcode)

138 Hagley Road
Edgbaston
Birmingham
B16 9PW

Patents ADP number (if you know it)

7442494002

6. Priority: Complete this section if you are
declaring priority from one or more earlier
patent applications, filed in the last 12 months.

Country

Priority application number
(if you know it)

Date of Filing
(day/month/year)

7. Divisionals, etc: Complete this section only if
this application is a divisional application or
resulted from an entitlement dispute (see note
f)

Number of earlier application

Date of filing
(day/month/year)

8. Is a Patents Form 7/77 (Statement of
inventorship and of right to grant of a patent)
required in support of this request? Answer 'Yes'

YES

if:
a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant,
or
c) any named applicant is a corporate body.
Otherwise answer NO (See note(d))

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form.
Do not count copies of the same document
Continuation sheets of this form

Description 5 + 5

Claim(s) 3 + 3

Abstract 0

Drawing(s) 0

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination (1)
(*Patents Form 9/77*)

Request for substantive examination
(*Patents Form 10/77*)

Any other documents
(*please specify*)

11. I/We request the grant of a patent on the basis of this application.

Signature
Barker Brettell
Barker Brettell

Date
15 OCTOBER 2003

12. Name and daytime telephone number of person to contact in the United Kingdom COLIN D KINTON Tel: 0121 456 1364

Warning
After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 01645 500505
 - Write your answers in capital letters using black ink or you may type them.
 - If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
 - If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
For details of the fee and ways to pay please contact the Patent Office.

FORMULATION FOR CORROSION AND SCALE INHIBITION

The present invention relates to a method of preventing or alleviating the problems associated with metal sulphide deposits and to novel
5 formulations for use in such a method.

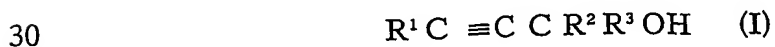
Tetrakis (hydroxyorgano) phosphonium salts (hereinafter THP⁺ salts); especially tetrakis (hydroxymethyl) phosphonium sulphate (hereinafter THPS) are widely used as metal sulphide dissolver/dispersers within
10 aqueous systems and especially those systems associated with oilfields.

When THPS is used in oilfields, it is typically applied in concentrations of up to 30%, together with an ammonium salt to improve performance. This combination of THPS and an ammonium salt, together with high
15 temperatures that can be experienced in oilfield applications, can be corrosive to mild steel and other metal components.

It is an aim of the present invention to ameliorate the above problems of corrosion by THP⁺ salts when used in aqueous systems.
20

Accordingly, the present invention, in a first aspect, provides a formulation for use in the treatment of corrosion and metal sulphide scale deposits in aqueous systems, said formulation comprising a THP⁺ salt (as hereinbefore defined), and a primary, secondary or tertiary alcohol
25 having an acetylenic bond in the carbon backbone.

Preferably, the acetylenic bond is adjacent to the hydroxyl group, said alcohol having the general formula (I):



wherein:

R^1 , R^2 and R^3 (which may be the same or different) each independently represent hydrogen, C_1 to C_8 alkyl or functionally substituted alkyl.

- 5 In a particularly preferred embodiment of the present invention, the alcohol is propargyl alcohol (2-propyn-1-ol), i.e. in formula (I), R^1 , R^2 and R^3 are each hydrogen.

- 10 The metal sulphide scale may be iron sulphide. Alternatively, the metal sulphide may be lead sulphide or zinc sulphide or a combination thereof. The iron sulphide may be Troilite (FeS) or Pyrite (FeS_2). Alternatively, the iron sulphide may be Mackinawite (Fe_9S_8) or Pyrrhotite (Fe_7S_8).

- 15 The anion of the THP^+ salt should be compatible with the aqueous system. Preferred anions include sulphate, chloride, phosphate, bromide, fluoride, carbonate, citrate, lactate, tartrate, borate, silicate, formate and acetate. The anion should make the THP^+ salt water-soluble.

- 20 The formulation may further include a surfactant. The surfactant is preferably a cationic surfactant, for example quaternary ammonium compounds, *N*-alkylated heterocyclic compounds or quaternised amidoamines. Alternatively, anionic, amphoteric or non-ionic surfactants may be used. Aminomethane phosphonates may replace ammonium salts in the aqueous system.

25

The formulation according to the invention is particularly useful in the prevention of corrosion of mild steel, copper and aluminium.

- 30 The present invention also provides, in a second aspect, a method for treatment of an aqueous system containing or in contact with a metal sulphide scale while concomitantly inhibiting the corrosion of surfaces in

contact with said aqueous system, which method comprises the addition to said aqueous system of a scale and corrosion inhibiting amount of a formulation in accordance with the first aspect of the invention.

5 The aqueous system is preferably one used in enhanced oil recovery. Alternatively, the aqueous system may be one used in industrial water systems, paper manufacturing systems and any aqueous system wherein corrosion caused by THP^+ salts occurs.

10 The present invention also provides, in a third aspect, a formulation consisting essentially of the reaction product of a THP^+ salt and an acetylenic alcohol in accordance with the first aspect of the present invention, wherein the ratio of said THP^+ salt to said acetylenic alcohol is between 1:1 and 750:1.

15

The formulation as described in the first aspect is preferably used in an effective amount up to 30% by weight as THP^+ . The amount used will vary by application but it may also be effectively used for low level applications e.g. 1 to 10000ppm as a THP^+ salt or in high level
20 applications as 1 to 30% as a THP^+ salt. In the second aspect THP^+ is preferably used in an effective amount of up to 30% by weight as a THP^+ salt, with the co-addition of an acetylenic alcohol in an effective amount of between 0.1 to 10000 ppm, relative to the volume of the system being treated.

25

The ratio of THP^+ to the acetylenic alcohol in the formulation is typically in the range 1:1 to 750:1, more preferentially 15:1 to 300:1, most preferably about 40:1.

30 The present invention will be illustrated, merely by way of example, as follows.

Example 1.

Blank Experiment: A 100ml solution of 20% THPS (26.6g TOLCIDE® PS75) and 1g ammonium chloride in synthetic seawater was placed in a 120ml screw-top jar. A pre-weighed mild steel coupon was immersed in the solution. The jar was then stored in a 50°C oven for 48 hours. After this time the coupon was cleaned by gentle scrubbing in water, washed with acetone and dried in the oven. The coupons were then reweighed and the corrosion rate calculated according to the equation:

10

$$\text{Rate} = \frac{K \times W}{A \times T \times d}$$

W = Weight loss in g (to 0.1mg)

15 A = Area in cm^2 (to 0.01 cm^2)

T = Time of exposure in hours

d = Density in g/cm^3

K is a constant defined by the units in which the corrosion rate is required. For example:

20

Units

Mpy - mils per year

K

3.45×10^6

25 This experiment was repeated with various levels of corrosion inhibitor added to the blank solution. The results are shown in Table A below:

TABLE A

Experiment	Corrosion Rate mpy
Blank	208
Blank + 5000ppm Inhibitor A	77
Blank + 5000ppm Inhibitor B	197
Blank + 5000ppm Inhibitor C	116
Blank + 5000ppm Inhibitor D	132
Blank + 5000ppm Inhibitor E	88
Blank + 5000ppm Inhibitor F	86
Blank + 5000ppm propargyl alcohol	33

5

Blank - A solution consisting of 20% THPS and 1% ammonium chloride in synthetic seawater.

Inhibitor A - Commercially available corrosion inhibitor comprising ethoxylated ammonium chloride, dibutyl thiourea and ethoxylated fatty acid.

10

Inhibitor B - Commercially available corrosion inhibitor comprising polyoxyethylene 2-ethylhexyl ether phosphate.

Inhibitor C - Oilfield corrosion inhibitor comprising fatty acids and Tall oil reaction products with diethylene triamine.

15 **Inhibitor D** - Commercially available corrosion inhibitor comprising a phosphonocarboxylic acid.

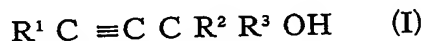
Inhibitor E - Standard commercially available oilfield corrosion inhibitor based on an amine alkoxylate.

20 **Inhibitor F** - Standard commercially available oilfield corrosion inhibitor comprising an amine ethoxylate and a quaternary ammonium chloride.

CLAIMS

1. A formulation for use in the treatment of corrosion and metal sulphide scale deposits in aqueous systems, said formulation comprising a THP⁺ salt (as hereinbefore defined) and a primary, secondary or tertiary alcohol having an acetylenic bond in the carbon backbone.

2. A formulation as claimed in Claim 1, in which the acetylenic bond is adjacent to the hydroxyl group, said alcohol having the general formula (I):



wherein:

R¹, R² and R³ (which may be the same or different) each independently represent hydrogen, C₁ to C₈ alkyl or functionally-substituted alkyl.

3. A formulation as claimed in Claim 1 or 2, in which the alcohol is propargyl alcohol.

4. A formulation as claimed in any one of Claims 1 to 3 in which the metal sulphide scale is iron sulphide.

5. A formulation as claimed in any one of Claims 1 to 3 in which the metal sulphide scale is lead sulphide.

6. A formulation as claimed in any one of Claims 1 to 3 in which the metal sulphide scale is zinc sulphide.

7. A formulation as claimed in any one of the preceding claims in which the THP⁺ salt comprises an anion selected from the group

7.

consisting of sulphate, chloride, phosphate, bromide, fluoride, carbonate, citrate, lactate, tartrate, borate, silicate, formate and acetate.

8. A formulation as claimed in any one of the preceding claims, said
5 formulation further including a surfactant.

9. A formulation as claimed in Claim 8 in which the surfactant is a cationic surfactant.

10 10. A formulation as claimed in Claim 9 in which the cationic surfactant is selected from the group consisting of quaternary ammonium compounds, N-alkylated heterocyclic compounds, quaternised amido-amines, and amino methane phosphonates.

15 11. A formulation as claimed in Claim 8 in which the surfactant is selected from the group consisting of anionic, amphoteric and non-ionic surfactants.

12. The use of a formulation as claimed in any one of the preceding
20 claims for treating corrosion of mild steel, copper or aluminium.

13. A method for treatment of an aqueous system containing or in contact with a metal sulphide scale while concomitantly inhibiting the corrosion of surfaces in contact with said aqueous system, which method
25 comprises the addition to said aqueous system of a scale and corrosion inhibiting amount of a formulation in accordance with any one of Claims 1 to 11.

14. A method according to Claim 13 in which the aqueous system is
30 used in enhanced oil recovery.

15. A method as claimed in Claim 13 in which the aqueous system is used in industrial water systems.
16. A method as claimed in Claim 13 in which the aqueous system is
5 used in paper manufacturing systems.
17. A formulation consisting essentially of the reaction product of a THP⁺ salt (as hereinbefore defined) and an acetylenic alcohol as claimed in any one of Claims 1 to 3, wherein the ratio of said THP⁺ salt and said
10 acetylenic alcohol is between 1:1 and 750:1.
18. A method as claimed in any one of Claims 13 to 16 in which the THP⁺ salt is added to the aqueous system in an effective amount of up to 30% by weight.
- 15 19. A formulation as claimed in any one of Claims 1 to 11 in which the ratio of the THP⁺ salt to the acetylenic alcohol is between 1:1 and 750:1.
20. A formulation as claimed in Claim 19 in which the ratio is between
20 15:1 and 300:1.
21. A formulation as claimed in Claim 19 or 20 in which the ratio is about 40:1.
- 25 22. A formulation substantially as described herein with reference to the accompanying example.
23. A method substantially as described herein with reference to the accompanying example.

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record.**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

☒ **BLACK BORDERS**

☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**

☐ **FADED TEXT OR DRAWING**

☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**

☐ **SKEWED/SLANTED IMAGES**

☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**

☐ **GRAY SCALE DOCUMENTS**

☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**

☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**

☐ **OTHER: _____**

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.